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US-A- 2 605 229  
US-A- 4 388 341  
US-A- 4 419 369

JOURNAL OF FOOD SCIENCE, vol. 48, 1983,  
pages 256-259,263, Institute of Food Tech-  
nologists Chicago, ILL, US; K.E. WEINGART-  
NER et al.: "Effects of calcium addition on  
stability and sensory properties of soy bev-  
erage"

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EP 0 234 773 B1

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## Description

The present invention relates to a method for producing a mineral-enriched protein composition which may be used in the production of foodstuffs, particularly liquid foodstuffs, such as dietary and nutritional beverages.

Protein isolates that are derived from vegetable protein sources, such as soybeans, have contributed substantially to the economic importance of these materials as a crop. Soy protein isolates, in particular, have proven to be a useful nutritional supplement in a variety of foods and beverages. A protein isolate can generally be characterized as a product resulting from the extraction, subsequent concentration and purification of proteinaceous material from a proteinaceous source, such as a vegetable protein material. Typically, the protein isolate on a moisture free basis will have a protein content which may range from 90% to 98% by weight.

The usefulness of soy protein isolates in the formation of foodstuffs such as beverages has, for the most part, been accomplished by the production of modified or enzymatically hydrolyzed isolates or the addition of materials such as surfactants to promote the dispersibility or suspendibility of the isolate in the particular type of aqueous medium that is used in preparation of the beverage.

An example of this type of protein product is described in U.S. Patent No. 4,378,378, in which a simulated milk product of improved suspension characteristics is produced by forming a slurry of a vegetable protein material and dairy whey, followed by reacting the slurry with a proteolytic enzyme. While protein isolates are generally dispersible in aqueous media, nevertheless it has been more difficult to employ these isolates in conjunction with certain vitamins and minerals that may be required, if a nutritionally complete beverage or drink, such as an infant formula, is to be produced. For example, fortification of a liquid product with calcium represents a particular problem, since most forms of calcium that are employed for calcium supplementation in nutritional beverages are relatively insoluble or poorly dispersible in aqueous media. These materials readily precipitate or settle from aqueous suspension, thereby providing the user with a drink that must be shaken relatively often to ensure adequate consumption of the minerals in the diet.

Dispersibility of a mineral-enriched protein composition has been described in U.S. Patent No. 4,214,996. In this, the mineral is chelated with an organic acid and a sugar to improve the dispersibility of the minerals in an aqueous medium such as a beverage. It is indicated that the product can be dried and reconstituted with good results.

An alternative approach to the dispersibility of specific mineral-fortifying substances, such as calcium phosphate, is set forth in U.S. Patent No. 2,605,229. This describes the production of a calcium phosphate gel which will remain dispersed in water when mixed therewith to yield a milky suspension simulating the dispersion of calcium phosphate in milk.

It would be desirable to develop a mineral-enriched protein composition of improved suspension characteristics in which no significant precipitation of the minerals or protein medium would occur, particularly upon prolonged storage of the beverage or liquid foodstuff to which the composition is added.

We have now discovered a method of producing a mineral-enriched protein composition, that can be used in the production of liquid foodstuffs, in which the mineral enriching components contained in the protein material substantially remain in suspension even during periods of prolonged storage.

The present invention is directed to a method of producing a mineral-enriched protein composition, which is used, for example, in the production of liquid foodstuffs, such as nutritional beverages and the like, which usually involve supplementation of the foodstuff with minerals and protein. The mineral-enriched protein composition is produced by a process comprising: forming a hydrated gel of an alkaline earth metal salt, such as a calcium salt, that is typically used for mineral enrichment or fortification of foodstuffs. This is followed by the addition of the hydrated gel of the alkaline earth metal salt to an aqueous slurry of an isolated vegetable protein material, such as a soy protein isolate, to form a mineral-enriched protein composition. The composition is then dewatered to provide a dried, mineral-fortified protein composition that can be used in the formation of liquid foodstuffs, such as nutritional beverages, requiring high levels of calcium or minerals for nutritional purposes.

It is preferred for the hydrated gel to be formed by reaction between an alkaline earth metal hydroxide and a mineral acid, particularly wherein the reaction is effected at a pH above 7, especially wherein the pH is from 7.5 to 9.5.

The process of the invention thus produces a mineral-enriched protein composition, which has improved suspension characteristics in liquid foodstuffs and overcomes problems associated with the suspension of mineral-fortifying materials in dietary beverages or nutritionally balanced, liquid drink products.

In a brief outline of the total process of the present invention, and for a complete understanding thereof, the vegetable protein material which forms a component of the composition is preferably produced by the following procedure. In this description, reference is made primarily to the use of soy protein, since this is the preferred starting material. However, it should be understood that the same process may be applied, *mutatis mutandis*, to other vegetable protein materials.

Soy protein isolate is typically produced from a starting material, such as defatted soybean material, from which the oil has been extracted to leave soybean meal or flakes. More specifically, the soybeans may be initially crushed or ground and then passed through a conventional oil expeller. It is preferable, however, to remove the oil contained in the soybeans by solvent extraction with aliphatic hydrocarbons, such as hexane or azeotropes thereof, and these represent conventional techniques employed for the removal of oil. The defatted, vegetable protein material or soybean flakes are then placed in an aqueous bath to provide a mixture having a pH of at least 6.5 and preferably between 7.0 and 10 in order to extract the protein. Typically, if it is desired to elevate the pH above 6.5 various alkaline reagents such as sodium hydroxide, potassium hydroxide or calcium hydroxide or other commonly accepted food grade alkaline reagents may be employed to elevate the pH. A pH of above 7 is generally preferred, since alkaline extraction facilitates solubilization of the protein. Typically, the pH of the aqueous extract of protein will be at least 6.5 and preferably from 7.0 to 10. The ratio by weight of the aqueous extractant to the vegetable protein material is usually from 20:1 to 1:1, a ratio of about 10:1 being preferred.

It is also desirable in obtaining the soy protein or other vegetable protein isolate used in the present invention, that an elevated temperature be employed during the aqueous extraction step to facilitate solubilization of the protein, although ambient temperatures are equally satisfactory if desired. The extraction temperatures which may be employed preferably range from ambient up to about 120 °F (48 °C), with a preferred temperature being about 90 °C (32 °C). The period for extraction is not critical and a period of from 5 to 120 minutes may be conveniently employed, with a preferred time of about 30 minutes. Following extraction of the vegetable protein material, the aqueous extract of protein is preferably stored in a holding tank or suitable container while a second extraction is performed on the insoluble solids remaining from the first aqueous extraction step. This improves the efficiency and yield of the extraction process by exhaustively extracting the protein from the residual solids from the first step.

The combined aqueous protein extracts from both extraction steps, normally having a pH of at least 6.5, or more preferably from 7.0 to 10, are then precipitated by adjustment of the pH of the extracts to, at, or near the isoelectric point of the protein to form an insoluble precipitate. The actual pH to which the protein extracts are adjusted will vary depending upon the vegetable protein material employed but, when a soy protein isolate is employed, this pH is typically between 4.0 and 5.0. The precipitation step may be conveniently carried out by the addition of a common food grade acidic reagent such as acetic acid, sulphuric acid, phosphoric acid, hydrochloric acid or any other suitable acidic reagent. Following precipitation of the protein, the solids level of the precipitated slurry is increased, for example by centrifugation or similar means, to concentrate the protein and remove, so far as possible, the whey or supernatant. The precipitated protein, in aqueous suspension, can then be employed in the production of the mineral-enriched protein composition as hereinafter described.

The vegetable protein isolate is then formed into an aqueous slurry for purposes of mineral enrichment as described below. Although the protein isolate slurry can be obtained directly from the isolation procedure as described above, in which the precipitated protein is still in the form of an aqueous suspension, it is equally possible in the present invention to employ as a starting material a dried protein isolate which is then dispersed into an aqueous medium to form an aqueous suspension. An essential aspect of the present invention, however, is the particular means for mineral fortification of the protein composition. It has been found, for example, that, if mineral fortification of the protein isolate takes place by the addition of a hydrated gel of the mineral fortifying material as compared to the addition of a dried, mineral supplement, a product of improved suspension characteristics is achieved. The improved suspension characteristics are also retained after drying of the fortified protein composition.

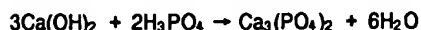
Typical alkaline earth materials which may be used for mineral fortification and which are considered to be essential for nutritional purposes include calcium and magnesium. Calcium has proven to be a particular problem insofar as fortification of protein supplements for liquid foodstuffs is concerned, since it is normally used at a higher fortification level than other minerals, in an aqueous medium such as an infant formula or nutritional beverage. For the most part this has been accomplished in the prior art by the dispersal of a dried calcium phosphate salt in the dried protein supplement, which, upon dispersion in an aqueous medium, still often results in settling of the mineral components during storage of the liquid foodstuff.

While the present invention is particularly directed towards the production of calcium-fortified, protein compositions of improved suspension characteristics, it is equally adaptable to other bivalent salts, such as

the alkaline earth metal salts, particularly magnesium salts, normally used for mineral fortification of foodstuffs. The exact means of forming the hydrated gel of the various alkaline earth metal salts is not critical to the practice of the present invention and these gels can be prepared by a variety of chemical reactions. Specifically, a hydrated calcium gel may be prepared by a reaction between calcium chloride and trisodium phosphate, to form a hydrated gel of tricalcium phosphate according to the following reaction.



Alternatively, a reaction between calcium hydroxide and phosphoric acid can be used with an equal degree of success to form a hydrated gel of tricalcium phosphate according to the following reaction.



The above reactions represent typical reactions for the production of a hydrated gel of an alkaline earth metal, such as calcium, that has been specifically found to improve the suspension characteristics of a vegetable protein material fortified with it.

When producing a hydrated gel of a calcium salt, it is preferable to employ the reaction set forth above in which calcium hydroxide is reacted with phosphoric acid, because no other salt (e.g. sodium chloride) is produced by this reaction. Typically, a dilute solution of calcium hydroxide is employed for the reaction with the phosphoric acid and, although the exact concentration of calcium is not critical, it is preferable that the calcium level in the solution should be about 0.1 to 3.0% by weight, preferably about 1.0% by weight. To this solution is added, in drop-wise fashion, concentrated phosphoric acid (e.g. 85% by weight) at a uniform and slow enough rate so that the pH of the reaction mixture is maintained above about 7. It is desirable to maintain the pH of the reaction medium to above 7 and preferably from 9.5 to 11.5 for purposes of producing the tricalcium phosphate which is the preferred material for purposes of calcium fortification of the protein composition. If the pH of the reaction mixture is allowed to fall below about 7 then the mono and dibasic forms of calcium phosphate are the primary products, and, while these may be used with equal success in the fortification of protein materials, and are covered by the present invention, it is preferred that the tricalcium phosphate be used since this material is the most stable form of calcium phosphate, for purposes of calcium fortification.

The reaction is allowed to proceed and a translucent hydrated gel of tricalcium phosphate begins to form. The hydrated gel, upon centrifugation, provides a gel having a solids content of less than about 10% by weight, and preferably from 7 to 10 percent by weight. It is this hydrated, translucent gel of tricalcium phosphate which has been found to provide a protein composition of improved suspension characteristics when it is used to provide mineral fortification of the protein composition. It is important that the hydrated gel should not be dried prior to addition to the protein slurry, since this has been found not to result in a mineral-fortified protein composition which has the desired suspension characteristics.

The pH of the protein slurry, which is produced as described above, is then adjusted to a desired value, e.g. from 6.5 to 7.5 and more preferably to from 6.8 to 7.0. As noted above, the protein slurry will typically have a solids level of from 5 to 20 percent by weight, more preferably from 12 to 20% by weight, and most preferably from 13 to 16% by weight, although the exact solids level is not critical insofar as the addition of the hydrated mineral gel is concerned. Generally speaking, the slurry of soy protein at this level of solids is considered to be pumpable, and therefore easily transferable in a continuous process.

The hydrated, mineral gel is then added to the protein slurry in an amount sufficient to provide a desired level of mineral fortification in the mineral-fortified protein composition, with the exact amount to be added to be dependent upon the degree of fortification desired. For example, in the case of a composition intended for adults, a level of about 1.5% by weight calcium based upon the protein solids in the composition is sufficient to meet normal daily requirements, whereas, in the case of a composition intended for infants or in the event one wants to simulate milk by providing a comparable calcium level, the level is usually from 2.7% to 3.5% by weight or even higher. Therefore, the exact amount of gel added is entirely dependent upon the degree of fortification desired, and the specific amount added is not intended to limit the present invention.

While it is preferable to employ a heating step as set forth below, a heating step is not essential to the present invention and can be omitted if desired, depending upon the type of protein composition desired. Typically a heating step is desirable for purposes of inactivating any undesirable enzyme inhibitors, such as trypsin inhibitors, and it is desirable to inactivate these inhibitors by the application of heat prior to the formulation of the protein composition. It is also not critical whether or not the heating step be employed before or after addition of the hydrated gel to the protein slurry. The protein slurry, containing the hydrated

gel added for purposes of fortifying the protein, is then preferably heated at a temperature of from 220 to 400°F (104 to 204°C), and more preferably at a temperature of from 260 to 310°F (126 to 154°C), preferably for a few seconds up to several minutes, and more preferably from 7 to 100 seconds. Preferably, heating is carried out in a jet cooker or similar apparatus, in which jets of steam intersect segments of the slurry in such a manner that the slurry is dynamically heated under conditions of both elevated temperature and pressure. Following heating of the slurry under these dynamic conditions of elevated temperature and pressure, the slurry containing the protein and minerals is typically ejected into a container of lower pressure, which causes volatilization of a portion of the water contained in the slurry with resultant cooling of the slurry to a temperature of about 150°F (65.5°C) or less.

The cooled slurry can then be dewatered by any type of drying procedure, but it is preferred to spray-dry the slurry to provide the most uniform mixture of the mineral fortifying salt and the protein and provide a product which has excellent dispersibility characteristics in aqueous media. The dried product has excellent suspension characteristics in a liquid foodstuff, and overcomes the separation problems normally associated with using mineral-fortified protein compositions in liquid foodstuffs, such as nutritional beverages.

The present invention is further illustrated with reference to the following Examples.

#### Example 1

A soy protein isolate was prepared in which 150 lb/hr (1.134 kg/minute) of defatted soybean flakes were fed to an extraction tank to which was added 1500 lb/hr (11.34 kg/minute) of water which was heated to 90°F (32°C). Sufficient calcium hydroxide was then added to adjust the pH of the mixture to 9.7. The soy flakes were extracted for a period of 30 minutes, after which the aqueous solution was separated from the extracted flakes by centrifugation. The first aqueous extract was held while the extracted flake residue was redispersed in 900 lb/hr (6.804 kg/minute) of water at a temperature of 90°F (32°C). The pH of the mixture at this point was 9.0.

A second aqueous extract from the flakes was obtained by centrifugation, and this was combined with the first aqueous extract. To the combined extracts was added 37% aqueous hydrochloric acid, to adjust the pH to a value of 4.5 and precipitate the protein. The precipitated protein was then centrifuged to remove excess liquid to a solids level of 24-28% by weight. The precipitated protein was then diluted with water to form a slurry having a solids level of 7.5% by weight. The pH of the slurry was adjusted to 6.6 by the addition of sodium hydroxide.

A hydrated gel of tricalcium phosphate was prepared by slowly adding an aqueous solution of calcium chloride (3.6% by weight) to an aqueous solution of trisodium phosphate (3.6% by weight) and reacting the mixture for 30 minutes. The pH of the mixture was above 9.5. A translucent, hydrated precipitate of tricalcium phosphate was formed, which was concentrated by centrifugation. The concentrated gel was then washed twice, each time with an equal weight of water, and again concentrated by centrifugation to a solids level of 6% by weight. The gel of tricalcium phosphate was then added to the slurry of soy isolate. The slurry, following addition of the gel, had a solids level of 7% by weight and a pH of  $7.0 \pm 0.05$ . The gel was added in an amount sufficient to provide a calcium level of 2.7% by weight of the protein solids on a dry basis, and the fortified slurry was allowed to equilibrate for 1 hour. The mineral-fortified slurry was then spray-dried to a moisture level of less than 5% by weight.

#### Example 2

A mineral-fortified slurry was prepared as described in Example 1, except that the washed residue or gel of tricalcium phosphate was dispersed in a solution of sodium hydroxide that contained sufficient sodium hydroxide to neutralize the slurry of acid-precipitated soy isolate to a pH value of 6.6 and to provide a slurry of the gel and isolate with a solids level of 7% by weight for spray-drying.

#### Example 3

A mineral-fortified protein slurry was produced as described in Example 1, except that, following addition of the gel of tricalcium phosphate to the protein slurry, the slurry was maintained at 90°C (32°C) for 10 minutes and homogenized at 2500 psi (178 kg/cm<sup>2</sup>) prior to spray-drying.

#### Example 4

A mineral-fortified protein slurry was produced as described in Example 1, except that the fortified

slurry had a solids level of 11% by weight prior to spray-drying.

#### Example 5

- 5 A mineral-fortified protein slurry was prepared as described in Example 2, except that the slurry had a solids level of 11% by weight prior to spray-drying.

#### Example 6

- 10 A mineral-fortified protein slurry was prepared as described in Example 1, except that a sufficient amount of the tricalcium phosphate gel was added to provide a calcium level of 5% by weight of the protein solids on a dry basis.

#### Example 7

- 15 A mineral-fortified protein slurry was prepared as described in Example 1, except that the slurry had a solids level of 5% by weight prior to drying. The fortified slurry was freeze-dried to a moisture level of 4% by weight.

#### Example 8

20 A mineral-fortified protein slurry was prepared as described in Example 1, except that the slurry contained a sufficient quantity of the gel to provide a calcium level of 20.0% by weight of the protein solids on a dry basis, and the fortified slurry had a solids level of 5% prior to spray-drying.

#### Example 9

- 25 A control sample was prepared in which a slurry of soy isolate was formed which was produced in accordance with Example 1, having a pH of 6.8 and a solids level of 7% by weight. To the slurry was added  
30 sufficient dried tricalcium phosphate to provide a calcium level of 2.7% by weight of the protein solids on a dry basis. The fortified slurry was then spray-dried as described in Example 1.

#### Example 10

- 35 Each of the calcium fortified isolates produced as described in Examples 1-9 was used to prepare an infant formula having the following formulation.

Ingredient	g/1000ml
Maltodextrin (15 D.E.)	69
Soybean Oil	34
Calcium-Fortified Soy Isolate (Examples 1-9)	24-26
Lecithin	1.0
Potassium Citrate	2.2
Sodium Chloride	0.5
Magnesium Chloride (MgCl <sub>2</sub> 6H <sub>2</sub> O)	0.53
Carrageenan	0.10
Water	867

- 50 The infant formula was prepared by the following procedure:

1. Disperse the carrageenan in the deionized water, and then solubilize the potassium citrate.
2. Disperse the mineral-fortified isolate (Examples 1-8) or control fortified isolate (Example 9) in the water solution.
3. Add the sodium and magnesium chloride and mix.
4. Check the pH of the mixture and adjust to 7.1 ± 0.1 if necessary.
5. Heat the mixture to 150 °F (66 °C).
6. Add the maltodextrin.
7. Add the soybean oil/lecithin preheated to 150 °F (66 °C).

8. Heat the mixture to 180°F (82°C).

9. Homogenize the mixture in a two stage homogenizer at 2,500 psi (176 kg/cm<sup>2</sup>) and 500 psi (35 kg/cm<sup>2</sup>).

10. Bottle the mixture, and retort at 255°F (124°C) for 11.5 minutes.

5 Table 1 sets forth an evaluation of each infant formula prepared using the protein compositions of Examples 1-9, for the stability of the suspension of the mineral in the formula, which was evaluated after 10 days storage.

Table 1

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Evaluation of Infant Formulas Prepared with Fortified Protein Compositions of Examples 1-9				
Example	pH of Formula	Mg of Calcium in 100g of Formula	Suspended Calcium	
			Mg/100g	% Total
1	6.8	85	85	100
2	6.8	56	48	82
3	6.8	73	73	100
4	6.8	54	51	94
5	6.8	60	43	72
6	6.9	64	64	100
7	6.8	65	42	65
8	6.9	53	27	51
9	6.7	60	12	20
(control)				

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The data in Table 1 show the improvement in suspension characteristics when the process of the present invention was used to prepare the mineral-fortified protein composition, which in turn was used in a nutritional beverage, such as an infant formula.

30

#### Example 11

A soy protein isolate was prepared in which 150 lb/hr (1134 g/minute) of defatted soybean flakes were fed to an extraction tank to which was added 1500 lb/hr (11.34 kg/minute) of water, which was heated to 90°F (32°C). Sufficient calcium hydroxide was added to adjust the pH of the mixture to 9.7. The soy flakes were extracted for a period of 30 minutes, after which the aqueous solution was separated from the extracted flakes by centrifugation. The first aqueous extract was held while the extracted flake residue was redispersed in 900 lb/hr (6.804 kg/minute) of water at a temperature of 90°F (32°C). The pH of the mixture at this point was 9.0.

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A second aqueous extract from the flakes was obtained by centrifugation and combined with the first aqueous extract. To the combined extracts was added 37% aqueous hydrochloric acid to adjust the pH to 4.5 and precipitate the protein. The precipitated protein was then centrifuged to remove excess liquid to a solids level of 24-28% by weight. The precipitated protein was then diluted with water to form a slurry having a solids level of 14.5% by weight. The pH of the slurry was adjusted to 6.8 by the addition of sodium hydroxide.

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A hydrated gel of tricalcium phosphate was prepared by slowly adding an aqueous solution of phosphoric acid (85% by weight) to an aqueous solution of calcium hydroxide (2.0% by weight), and the mixture was reacted for 30 minutes. A translucent, hydrated precipitate of tricalcium phosphate was formed, which was concentrated by centrifugation. The concentrated gel of tricalcium phosphate was then added to the slurry of soy isolate. The slurry, following addition of the gel, had a solids level of 14% by weight and a pH of 7.0 ± 0.05. The gel was added in an amount sufficient to provide a calcium level of 2.6% by weight of the protein solids on a dry basis and the fortified slurry was allowed to equilibrate for 1 hour.

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The calcium-fortified slurry was then passed through a jet cooker at a pressure of 85 psig (5.98 kg/cm<sup>2</sup>). The steam heated the slurry in the jet cooker to a temperature of 310°F (154°C). After 8-10 seconds, progressive portions of the heated slurry were discharged into a receiver at below atmospheric pressure. The mineral-fortified slurry was then spray-dried to a moisture level of less than 5% by weight.

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#### Example 12

A mineral-fortified protein slurry was prepared as described in Example 11, except that 1.0% by weight aqueous solutions of calcium hydroxide and phosphoric acid were used to prepare the mineral gel for addition to the isolate. The gel was added in this Example in an amount sufficient to provide a calcium level of 2.7% by weight of the protein solids.

#### Example 13

Each of the isolates obtained as described in Examples 11-12 was used to prepare infant formulas as generally described in Example 10. Table 2, set forth below, contains an evaluation of each infant formula prepared using the protein compositions of Examples 11-12, for the stability of the suspension of the mineral in the formula. A control sample was used for comparison, which was produced by adding dried tricalcium phosphate to a nonfortified isolate.

Table 2

Evaluation of Infant Formulas Prepared with Fortified Protein Composition of Examples 11-13				
Example	pH of Formula	Mg of Calcium in 100g of Formula	Suspended Calcium	
			Mg/100g	% Total
11	6.8	67	67	100
12	6.8	65	62	94
control	6.8	75	15	20

It is apparent that the calcium-fortified isolate prepared according to the present composition exhibited better performance in the infant formulas, insofar as suspension of the calcium in the formulas is concerned.

#### Claims

1. A method of producing a mineral-enriched protein composition for use in the production of foodstuffs, which comprises:
  - (a) forming a hydrated gel of an alkaline earth metal salt suitable for mineral enrichment;
  - (b) adding said hydrated gel to an aqueous slurry of an isolated vegetable protein material to form a mineral-enriched protein composition; and
  - (c) dewatering said composition.
2. A method according to Claim 1, wherein the alkaline earth metal salt is a calcium salt.
3. A method according to Claim 2, wherein the salt is a calcium phosphate.
4. A method according to Claim 2, wherein the salt is tricalcium phosphate.
5. A method according to any one of Claims 2 to 4, wherein the dewatered composition has a calcium level of at least 1.5% by weight of the protein solids.
6. A method according to Claim 5, wherein the dewatered composition has a calcium level of at least 2.7% by weight of the protein solids.
7. A method according to any one of the preceding Claims, wherein the hydrated gel is formed by reaction between an alkaline earth metal hydroxide and a mineral acid.
8. A method according to Claim 7, wherein the reaction is effected at a pH above 7.
9. A method according to Claim 8, wherein the pH is from 7.5 to 9.5.
10. A method according to any one of Claims 7 to 9, wherein the mineral acid is phosphoric acid.
11. A method according to any one of the preceding Claims, wherein the slurry has a solids level of from 5



to 20% by weight.

12. A method according to any one of the preceding Claims, wherein the slurry has a pH of from 6.5 to 7.5.
- 5 13. A method according to any one of the preceding Claims, wherein the hydrated gel has a solids level which is less than 10% by weight.
14. A method according to any one of the preceding Claims, wherein dewatering is carried out by spray-drying.
- 10 15. A method according to any one of the preceding Claims, wherein the vegetable protein material is soy.
16. A method according to any one of the preceding Claims, wherein the aqueous slurry of vegetable protein material is heated to a temperature of from 220 to 400 °F (104 to 204 °C) for a period of time
- 15 sufficient to inactivate enzyme inhibitors in said protein.
17. A foodstuff containing a mineral-enriched protein composition produced by a method according to any one of the preceding Claims.
- 20 18. A foodstuff according to Claim 17, which is a liquid beverage.

#### Revendications

- 25 1. Une méthode de production d'une composition de protéines enrichie en produits minéraux pour une utilisation dans la production de produits alimentaires, qui comprend:
  - (a) la formation d'un gel hydraté d'un sel de métal alcalino-terreux adapté à un enrichissement en produits minéraux;
  - (b) l'addition dudit gel hydraté à une bouillie aqueuse d'une matière protéinique végétale isolée pour former une composition de protéines enrichie en produits minéraux et
  - 30 (c) l'égouttage de ladite composition
2. Une méthode suivant la Revendication 1, dans laquelle le sel de métal alcalino-terreux est un sel de calcium.
- 35 3. Une méthode suivant la Revendication 2, dans laquelle le sel est un phosphate de calcium.
4. Une méthode suivant la Revendication 2, dans laquelle le sel est le phosphate tricalcique.
5. Une méthode suivant l'une quelconque des Revendications 2 à 4, dans laquelle la composition
- 40 égouttée a un taux en calcium d'au moins 1.5% en poids des matières protéiniques à l'état solide.
6. Une méthode suivant la Revendication 5, dans laquelle la composition égouttée a un taux en calcium d'au moins 2.7% en poids des matières protéiniques à l'état solide.
- 45 7. Une méthode suivant l'une quelconque des Revendications précédentes, dans laquelle le gel hydraté est formé par réaction entre un hydroxyde de métal alcalino-terreux et un acide minéral.
8. Une méthode suivant la Revendication 7, dans laquelle la réaction est effectuée à un pH supérieur à 7.
- 50 9. Une méthode suivant la Revendication 8, dans laquelle le pH est de 7.5 à 9.5.
10. Une méthode suivant l'une quelconque des Revendications 7 à 9, dans laquelle l'acide minéral est de l'acide phosphorique.
- 55 11. Une méthode suivant l'une quelconque des Revendications précédentes, dans laquelle la bouillie a un taux en matières solides de 5 à 20% en poids.
12. Une méthode suivant l'une quelconque des Revendications précédentes, dans laquelle la bouillie a un

pH de 6.5 à 7.5.

13. Une méthode suivant l'une quelconque des Revendications précédentes, dans laquelle le gel hydraté a un taux en matières solides qui est inférieur à 10% en poids.
14. Une méthode suivant l'une quelconque des Revendications précédentes, dans laquelle l'égouttage est effectué par séchage par pulvérisation.
15. Une méthode suivant l'une quelconque des Revendications précédentes, dans laquelle la matière protéinique d'origine végétale est du soja.
16. Une méthode suivant l'une quelconque des Revendications précédentes, dans laquelle la bouillie aqueuse de matière protéinique d'origine végétale est chauffée jusqu'à une température comprise entre 220 et 400° F (104 à 204° C) pendant une période suffisante pour inactiver les inhibiteurs d'enzymes dans ladite protéine.
17. Un produit alimentaire contenant une composition de protéines enrichie en produits minéraux, produit par une méthode suivant l'une quelconque des Revendications précédentes.
18. Un produit alimentaire suivant la Revendication 17, qui est un breuvage.

#### Patentansprüche

1. Verfahren zum Herstellen einer mit Mineralien angereicherten Proteinmischung für die Verwendung bei der Herstellung von Nahrungsmitteln, bei welchem
  - (a) ein hydratisiertes Gel eines für die Mineralanreicherung geeigneten Erdalkalimetallsalzes gebildet wird,
  - (b) das hydratisierte Gel zwecks Bildung einer mit Mineralien angereicherten Proteinmischung einem isolierten Pflanzenproteinmaterial zugesetzt wird und
  - (c) diese Mischung entwässert wird.
2. Verfahren nach Anspruch 1, worin das Erdalkalimetallsalz ein Calciumsalz ist.
3. Verfahren nach Anspruch 2, worin das Salz ein Calciumphosphat ist.
4. Verfahren nach Anspruch 2, worin das Salz Tricalciumphosphat ist.
5. Verfahren nach irgendeinem der Ansprüche 2 bis 4, worin die entwässerte Mischung einen Calciumgehalt von zumindest 1,5 Gew.-% der Proteinfeststoffe besitzt.
6. Verfahren nach Anspruch 5, worin die entwässerte Mischung einen Calciumgehalt von zumindest 2,7 Gew.-% der Proteinfeststoffe besitzt.
7. Verfahren nach irgendeinem der vorhergehenden Ansprüche, worin das hydratisierte Gel durch Umsetzung zwischen einem Erdalkalimetallhydroxid und einer Mineralsäure hergestellt wird.
8. Verfahren nach Anspruch 7, worin die Umsetzung bei einem pH-Wert von mehr als 7 vorgenommen wird.
9. Verfahren nach Anspruch 8, worin der pH-Wert 7,5 bis 9,5 beträgt.
10. Verfahren nach irgendeinem der Ansprüche 7 bis 9, worin die Mineralsäure Phosphorsäure ist.
11. Verfahren nach irgendeinem der vorhergehenden Ansprüche, worin die Aufschlämmung einen Feststoffgehalt von 5 bis 20 Gew.-% besitzt.
12. Verfahren nach irgendeinem der vorhergehenden Ansprüche, worin die Aufschlämmung einen pH-Wert von 6,5 bis 7,5 besitzt.

**EP 0 234 773 B1**

13. Verfahren nach irgendeinem der vorhergehenden Ansprüche, worin das hydratisierte Gel einen Feststoffgehalt von weniger als 10 Gew.-% besitzt.
14. Verfahren nach irgendeinem der vorhergehenden Ansprüche, worin das Entwässern durch Sprühtrocknen vorgenommen wird.
15. Verfahren nach irgendeinem der vorhergehenden Ansprüche, worin das pflanzliche Proteinmaterial Sojamaterial ist.
16. Verfahren nach irgendeinem der vorhergehenden Ansprüche, worin die wässrige Aufschlämmung des Pflanzenproteinmaterials während einer zum Inaktivieren von Enzym-inhibitoren in diesem Protein ausreichenden Zeit auf eine Temperatur von 220 bis 400 ° F (104 bis 204 ° C) erhitzt wird.
17. Nahrungsmittel, welches eine mit Mineralien angereicherte Proteinmischung enthält, die nach einem Verfahren gemäß irgendeinem der vorhergehenden Ansprüche hergestellt worden ist.
18. Nahrungsmittel nach Anspruch 17, welches ein flüssiges Getränk ist.

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